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OHIO STATE UNIVERSITY

Research Foundation
1960 Kenny Road
Columbus, OH 43210-1063

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July 30, 1993

Defense Technical Information Center
Building 5, Cameron Station
Alexandria, Virginia 22304-6145

Dear Colleague:

Enclosed are 12 **copies of the report**

"Interdisciplinary Research on Wear of Materials"

prepared under Contract/Grant No. N00014-89-J-1234**.**

We have enjoyed working with you on this project and trust that this report is satisfactory. We look forward to the opportunity of working with you again in the future.

Sincerely,

Joy Ann Fischer

Joy Ann Fischer
Director, Editorial and Printing Services

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Rigney/Rundle - RF

FY92 End of Fiscal Year Letter
and Final Technical Report
(01 Oct 1991 - 30 Sep 1992)

ONR CONTRACT INFORMATION

Contract Title: Interdisciplinary Research on Wear of Materials

Performing Organization: The Ohio State University

Principal Investigator: D. A. Rigney

Contract Number: N00014-89-J-1234

R & T Project Number: 4316014

ONR Scientific Officer: P. P. Schmidt, ONR Code 3312

Enclosure (1)

93-8-3-192

FISCAL YEAR AND FINAL REPORT

on

INTERDISCIPLINARY RESEARCH ON WEAR OF MATERIALS

to

The Office of Naval Research

July 28, 1993

D. A. Rigney
Materials Science and Engineering
The Ohio State University
116 West 19th Avenue
Columbus, OH 43210-1179

W. A. Glaeser
Battelle Columbus Labs.
505 King Avenue
Columbus, OH 43201-2693

A. DESCRIPTION OF THE RESEARCH GOALS AND APPROACH

This interdisciplinary research was designed to provide complementary information on materials subjected to lubricated and unlubricated sliding contact. With the aid of changes in test environment, surface activation, ion implantation, electron microscopy, diffraction analysis, surface analysis (Auger and laser Raman), and fracture mechanics, basic mechanisms of wear are being related to plastic flow, stress intensity and surface chemistry. The three main thrusts have been lubricated sliding, unlubricated sliding and fracture considerations.

B. PROGRESS AND SIGNIFICANT RESULTS IN THE PAST YEAR

Lubricated Wear Surface Studies

A number of in situ wear experiments using the pin-on-disk apparatus operating in an Auger vacuum chamber have continued. Steel sliding against steel lubricated with ZDDP (zinc dialkyldithiosulfate) were involved (AISI 1040 steel pin and AISI 1040 steel disk in the annealed condition). The ZDDP lubricant was in solution (saturated solution in dodecane). The objective of these experiments was to determine the surface chemistry involved in boundary lubrication with ZDDP using surface analysis by Auger and ESCA spectroscopy in the wear track as it develops. In addition, the effect of continuous sliding contact on the chemistry of the boundary film was explored.

The steel disk was pre-run in air with the lubricant solution applied to the disk surface. With the pin located in the wear track, the apparatus was inserted in the Auger vacuum chamber and the vacuum established. The pin-on-disk was run for a number of revolutioins and the lubricating film analyzed periodically to record the surface chemistry as a function of disk revolutions.

The native oxide on steel was found to be an essential ingredient in the lubricating process. A complex reaction between the mixed alkyl sulfides in ZDDP and the surface oxide resulted in a sulfide-oxide mixed film approximately 6 to 10 nm thick. A contiguous oxide phase next to the steel substrate was not found in the wear tracks. A thin hydrocarbon film from the in-air operation was found intact in the vacuum chamber and influenced in situ friction for a short time until it was worn off.

These experiments show that lubrication is accomplished by the formation of a mixed sulfide-oxide not detectable by light microscopy. Oxide in the steel surface is an essential component in this reaction-lubrication process. Depletion of the oxide kills the lubricating action.

Wear debris attrition experiments by ball milling were carried out with an iron-nickel alloy. ZDDP in dodecane solution was used as the lubricating medium in the ball mill. Debris products from these experiments were analyzed in the transmission electron microscope. Many sub-micron size particles were thin enough to transmit electrons so that dislocation structures could be examined. Very small crystalline particles were found associated with the larger electron transmitting particles. These particles, one to five nm in size, showed up by forming Moire patterns. Possible nucleation of nickel crystals during the high strain deformation were considered as an explanation for the nanometer size particles. These experiments have continued to show that attrition of wear debris in the presence of a lubricant produces a much finer product than without lubricant. Reduction of wear debris to a very fine size makes it more benign in tribological applications.

Dry Sliding

A major contribution arising from research sponsored earlier by ONR (and described in earlier reports) was identification of a sequence of events commonly involved in the sliding wear of simple metals. Characterization of samples and debris by SEM, EDS, WDS, AES, XRD, TEM/STEM, microhardness, etc., showed that adhesive transfer, plastic deformation and mechanical mixing were all important. The results of that work have guided the more recent activities during this project, in particular the work of Venkatesan (2) which used Co-56 produced by activation in a Van de Graaff generator to study friction and wear transitions in steels and the work of Zhang (3) involving ion implantation.

(a.) Mild and Severe Wear of Steel:

The classic work of Welsh (4,5) helped to guide our work on mild and severe wear of 1045 steel. The role of transfer was investigated by by making one member of the sliding pair radioactive by exposure to high-energy protons in our Van de Graaff generator. The resulting Co-56 could be detected easily in small concentrations. Significant transfer was not detected in these steel samples. Therefore, transfer was not a principal factor in the unlubricated sliding behavior of these specimens in either air or vacuum for the test conditions used.

The mild and severe wear rates in air differed by about two orders of magnitude. Wear rates in vacuum were less than severe wear rates in air, in contrast to expectations based on the assumption that mild wear is more likely when oxide films can be produced more easily. Magnetite was found in all wear debris samples. A small amount of alpha Fe_2O_3 was found in the debris from mild wear tests. Alpha iron was found in debris from severe wear tests in air and from tests in vacuum. Patches of extreme hardness were found on wear surfaces after tests in vacuum, despite the low sliding speeds used. A smooth coating developed on the surface of the fixed sample when mild conditions prevailed. This special coating should be studied further, for both unlubricated and lubricated sliding conditions.

(b.) Ion Implantation Studies:

This part of the project was designed to provide new information on the effects of ion implantation on surface (adhesion) and subsurface (deformation) effects during unlubricated sliding of metals. The base materials are copper and iron, and the implanted species are also copper and iron. Thus, in some samples there should only be structural changes caused by implantation (e.g., Fe into Fe, Cu into Cu), while in others there will be composition changes as well (e.g., Cu into Fe, Fe into Cu). Copper and iron were chosen because of the considerable amount of literature available for comparison of results from testing of these metals. Also, their hardness values and deformation behavior are different, the OSU group has experience with both metals, and the phenomenon of selective transfer, extensively studied in the former USSR, usually involves both Cu and Fe.

The work was originally designed to address questions raised at a workshop at Argonne National Laboratory in 1988 (1), in particular questions associated with separating surface and subsurface effects. Recognizing that these may interact in ways which make them difficult to separate, we have attempted to use ion implantation of samples tested in vacuum in a pin/disk sliding system. Because geometric effects are known to be important, we are using both Cu/Fe and Fe/Cu configurations (pin/disk). There are thus 36 different combinations of pin/disk for each implanted thickness used, because each component can be Cu, Fe, Cu(Cu), Fe(Fe), Cu(Fe) or Fe(Cu), where the species in parentheses is the implanted species.

The graduate student working on this project passed her General Examination during Winter Quarter, 1991. Since then, she has been working full-time on her research. She has had to surmount a number of difficult problems which slowed initial progress. For example, the first set of implanted specimens was contaminated with unacceptable amounts of carbon and cadmium. The student found that the source of the carbon contamination was the furnace used earlier for annealing the specimens. She also traced the Cd contamination to plated screws used in the implantation chamber at Mound Laboratories, which is providing implantation services. These problems were identified and solved by the student herself. The student is also becoming proficient with several scanning and transmission electron microscopes and with specimen preparation for those instruments, and she has begun a systematic literature review for her project.

Mound Laboratories will implant two series of samples. These will use different accelerating voltages and fluences to give different implantation depths and concentration profiles.

Modeling, Including Fracture Considerations

This part of the research continued along two main lines, one emphasizing fracture modeling and testing of brittle materials and the other considering the unusually high energy involved in the wear of metals. Linear elastic fracture mechanics was used to model brittle materials under combined shear and compression, typical conditions of loading during sliding. The effects of rubbing of crack faces was incorporated. Experiments used a new slotted-disk test developed at Battelle to obtain a desired mix of compression and shear. The work done was comparable to that found in tensile tests. For metals, the focus was on the inefficiency of the sliding wear process, in which much work is done for a given amount of debris generation. The emphasis was on a newly defined wear energy (6).

C. RESEARCH PLANS FOR THE NEXT FISCAL YEAR

1. The following work was planned, but it was postponed when the proposal to continue the interdisciplinary project was not accepted:

(a.) Further work on lubricated wear would involve AES and Raman in situ sliding experiments with an emphasis on the effect of oxide thickness and structure on boundary lubrication (stearic acid and ZDDP). Ball mill experiments with surfactants would be used to generate powders for comparison with wear debris. A new thrust would involve application of electrochemistry to the study of oxidation and chemiabsorption of organic species in systems with boundary lubrication. A quick response potentiostat attached to an AES system would be used.

(b.) Available wear maps would be used to select conditions for mild and severe wear of iron and selected steels. The focus would be on the role of oxide films (formation, structure, control), especially in mild wear. Tests would be both in air and vacuum. Structural and chemical characterization would use techniques described in our previous work but augmented by XPS, Raman and STM/AFM.

(c.) Our recent work on fracture aspects of sliding wear has highlighted the unusually high energy involved in wear processes. This newly defined wear energy would be the focus of the analysis. The fracture mechanics work would be extended to a crack <u>growth</u> model and to environmentally assisted fracture.

2. Implanted specimens received from Mound Laboratories will be tested in vacuum, using both short and long sliding distances. This will allow checking behavior before and after any mild to severe transition which may appear. Note: This work continued under a new project number, N00014-92-J-1608, Continuing Research on Friction and Wear of Materials.

Report References:

1. Workshop on Wear Modeling, Argonne National Laboratory, Argonne Laboratory, Argonne, IL, June 16-17, 1988.

2. S. Venkatesan and D. A. Rigney, Sliding Friction and Wear of Plain Carbon Steels in Air and Vacuum, Wear 153(1992)163-178.

3. L. H. Zhang, Ph.D. project in progress, The Ohio State University.

4. N. C. Welsh, Phil. Trans. Roy. Soc. A257(1965)31-49.

5. N. C. Welsh, Phil. Trans. Roy. Soc. A257(1965)51-70.

6. A. R. Rosenfield, Wear and Fracture Mechanics: Are They Related?, Scripta Met. et Mat. 24(1990)811-814.

D. LIST OF PUBLICATIONS/REPORTS/PRESENTATIONS

1. Papers Published in Refereed Journals

S. Venkatesan and D. A. Rigney, Sliding Friction and Wear of Plain Carbon Steels in Air and Vacuum, invited for special volume of Wear 153(1992)163-178.

S. M. Kuo and D. A. Rigney, Sliding Behavior of Aluminum, Materials Sci. and Engin., A157(1992)131-143.

D. A. Rigney, Some Thoughts on Sliding Wear, Wear 152(1992) 187-192.

D. A. Rigney, The Role of Characterization in Understanding Debris Generation, in Wear Particles, Proceedings, 18th Leeds/Lyon Symposium, Lyon, France, eds. D. Dowson et al., pp. 405-412, 1992.

D. A. Rigney, R. Divakar and S. M. Kuo, Deformation Substructures Associated with Large Plastic Strains, invited for Viewpoint Set by N. Hansen, Scripta Met. et Mat. 27 (1992)975-980.

M. Sato, P. M. Anderson and D. A. Rigney, Rolling-Sliding Behavior of Rail Steels, accepted for International Conference on Wear of Materials, April, 1993.

W. A. Glaeser, Ball Mill Simulation of Wear Debris Attrition, Wear Particles: from the Cradle to the Grave, Proceedings 18th Leeds/Lyon Symposium on Tribology, D. Dowson et al., eds., Elsevier, 1992, pp. 515-523.

W. A. Glaeser, D. Baer and M. Engelhardt, In Situ Wear Experiments in the Scanning Auger Spectrometer, accepted for International Conference on Wear of Materials, April, 1993.

A. R. Rosenfield, Slow Crack Growth in glass in Combined Mode I and Mode II Loading, with D. K. Shetty, Scripta Met. et Mat. 25 (1991)997-1002.

A. R. Rosenfield, Combined Mode I-Mode III Fracture Toughness of a Spheroidized 1090 Steel, with M. Manoharan and J. P. Hirth, Acta Met. et Mat. 39(1991)1203-1210.

A. R. Rosenfield, Discussion of 'Wear Mode Map of Ceramics' by K. Hokkirigawa, Wear 151(1991)403-406.

2. Non-Refereed Publications and Published Technical Reports

A. R. Rosenfield, Fracture Toughness of Ceramic bonds Using a Chevron Notch Disk Specimen, with B. S. Majumdar, ASTM STP 1172 (1992)63-73.

A. R. Rosenfield, Nodular Cast Iron for Transport of Spent Nuclear Waste, Materials Development in Rail, Tire, Wing, Hull Transportation, Associazione Italiana di Metallurgia, 1992, 2.265-2.274.

3. Presentations

a. Invited

D. A. Rigney, Detroit Section, TMS-AIME, .earborn, MI, Nov. 4, 1991.

D. A. Rigney, First Plenary Lecture, The Roles of Hardness in the Sliding Behavior of Materials, Int'l. Conf. on New Materials and Technologies in Tribology, Minsk, Belarus, Oct. 6-9, 1992.

b. Contributed

W. A. Glaeser, Simulation of Wear Debris in a Ball Mill, Navy Tribology Workshop, U. S. Naval Academy, May 11-13, 1992.

A. R. Rosenfield, Nodular Cast Iron for Transport of Spent Nuclear Waste, Materials Development in Rail, Tire, Wing, Hull Transportation, Associazione Italiana di Metallurgia, Genoa, Italy, Nov., 1992.

4. Books (and sections thereof)

D. A. Rigney, The Role of Characterization in Understanding Debris Generation, in Wear Particles, Proceedings, 18th Leeds/Lyon Symposium, Lyon, France, eds. D. Dowson et al., 1992, pp. 405-412.

W. A. Glaeser, Ball Mill Simulation of Wear Debris Attrition, Wear Particles: from the Cradle to the Grave, Proceedings 18th Leeds/Lyon Symposium on Tribology, D. Dowson et al., eds., Elsevier, 1992, pp. 515-523.

A. R. Rosenfield, Fracture Characterization of 'Super Clean' Rotor Steel Using Advanced Techniques, with J. P. Hirth and S. Ragavachary, Superclean Rotor Steels, ed. R. I. Jaffee, 1991, pp. 321-327.

A. R. Rosenfield, Investigation of the Bases of the K_{IR} Curve, with D. E. McCabe, R. K. Nanstad, C. W. Marschall, and G. R. Irwin, ASTM PVP 213, Pressure Vessel Integrity, 1991, pp. 141-148.

A. R. Rosenfield, Ballistic Performance of Selected Ceramic-Cored Targets Impacted by a Shaped Charge Jet, with C. W. Marschall, W. H. Duckworth, and R. A. Stein, Combat Vehicle Survivability Symposium, U. S. Army Tactical Automotive Command, Vol. 2, 1992, pp. 143-152 (abstract only; paper classified secret).

Enclosure (2)

E. LIST OF HONORS/AWARDS

Name of Person Receiving Award	Recipient's Institution	Name, Sponsor and Purpose of Award
D. A. Rigney	The Ohio State U.	First Plenary Lecture Int'l. Conf. on New Mat'ls. and Technologies in Tribology, Minsk, Belarus, Oct. 6-9, 1992.

Enclosure (3)

F. PARTICIPANTS AND THEIR STATUS

1. Dr. D. A. Rigney, Professor, Materials Science and Engineering, The Ohio State University, Project P.I.

2. L. H. Zhang, Graduate Research Assistant, Ph.D. candidate; passed General Examination for candidacy for Ph.D.--working full-time on this project. Expected date of receiving degree: 12/93.

3. W. A. Glaeser, Battelle Columbus Laboratories, P.I. for Project Sub-Contract.

4. Dr. A. R. Rosenfield, Battelle Columbus Laboratories, Project Sub-Contract, fracture mechanics contributions and wear modeling.

5. Dr. D. R. Baer, Battelle Northwest, Project Sub-Contract, surface chemistry contributions.

6. S. Venkatesan , Graduate Research Assistant, received Ph.D., August, 1991, with dissertation on sliding of steels, using activation to determine the effect of transfer on friction and wear transitions; became post-doctoral researcher with Dr. B. Bhushan, Dept. of Mechanical Engineering, The Ohio State University, for one year, working on friction of silicon and other problems related to the tribology of computer systems.

G. OTHER SPONSORED RESEARCH DURING FY92

1. D. A. Rigney, Selective Transfer in Tribology, NSF, Engineering Division (Surface Engineering and Tribology), SGER program, $35,000, 7/1/91-9/14/93, for one graduate student.

2. D. E. Kim and D. A. Rigney, Surface Modification for Controlling Friction and Wear, OSU-CMR (Center for Materials Research) sponsored project, Office of Research, The Ohio State University, $44,044, Jan 1, 1992-June 30, 1993, for one graduate student in MSE Dept. and one in Mech. E. Dept.

3. W. A. Glaeser, Wear of Bearing Materials for Refrigeration Compressors, Copeland Corp., Sidney, OH, June, 1992-May, 1993.

4. A. R. Rosenfield:
    - (a.) Armor/anti-armor program, DARPA/DOE, 1988-present.
    - (b.) Ceramic Engine program, subcontract from Allison (DOE), 1991-1992.
    - (c.) Heavy Section Steel Technology Support Program (DOE/MMES), 1979-present.
    - (d.) Overhead transmission line program, EPRI, 1991-1992.
    - (e.) Mortar degradation, Gas Research Institute, 1990-present.

H. SUMMARY OF FY92
PUBLICATIONS/PATENTS/PRESENTATIONS/HONORS/PARTICIPANTS
(Number Only)

		ONR	non ONR
a.	Number of Papers Submitted to Referred Journal but not yet published:	0	0
b.	Number of Papers Published in Refereed Journals:	8	11
c.	Number of Books or Chapters Submitted but not yet Published:	0	0
d.	Number of Books or Chapters Published:	2	3
e.	Number of Printed Technical Reports & Non-Referred Papers:	1	1
f.	Number of Patents Filed:	0	0
g.	Number of Patents Granted:	0	0
h.	Number of Invited Presentations at Workshops or Prof. Society Meetings:	2	0
i.	Number of Contributed Presentations at Workshops or Prof. Society Meetings:	1	1
j.	Honors/Awards/Prizes for Contract/Grant Employees: (selected list attached)	1	0
k.	Number of Graduate Students and Post-Docs Supported at least 25% this year on contract grant:	1	3
	Grad Students: TOTAL	1	3
	Female	1	0
	Minority	1	1
	Post Doc: TOTAL	0	0
	Female	0	0
	Minority	0	0
l.	Number of Female or Minority PIs or CO-PIs		
	New Female	0	0
	Continuing Female	0	0
	New Minority	0	0
	Continuing Minority	0	0

Enclosure (4)